Derivatization and Spectroscopic Procedures

Typical medium Scale Procedure :

To 84 mg of (*S*)-(+)-2-amino-1-propanol dissolved in 2 mL ethanol were added 2.2 mL of a 0.5 M aqueous solution of sodium hydroxide in a round bottom flask equipped with a magnetic stirring bar. After 5 minutes stirring, 500 mg of 2-(bromomethyl)quinoline in 3.5 mL of ethanol were poured slowly into the reaction with 5 mL of a 0.5 M aqueous solution of sodium hydroxide. After one hour, 20 mL of methylene chloride was added. The organic layer was collected, dried over potassium carbonate and evaporated to dryness. The crude product was dissolved in 2 mL of methylene chloride and passed through a SilicaSPE column (Varian, Inc.). The byproducts were eluted with 300 mL methylene chloride prior to eluting the product with ethyl acetate. The ethyl acetate was collected and evaporated.

A 0.3 mM methanolic solution of the free ligand was prepared. One equivalent of each $Cu(ClO_4)_2$ and NH_4SCN were added and the CD spectrum recorded.

Typical small Scale Procedure :

To 0.03 μL of an aqueous solution being 0.5 M in (*D*)-tryptophan and 1.0 M in sodium hydroxide was added 0.03 μl of a 1.0 M ethanolic solution of 2-(bromomethyl)quinoline. After 6 minutes of centrifugation another 0.03 μL of a 1.0 M aqueous solution of sodium hydroxide were added. The reaction mixture was centrifuged for 6 minutes. Methylene chloride (2 mL) was added to the crude reaction and the organic layer dried over anhydrous potassium carbonate. Evaporation of the solvent yielded the crude product which was taken up in 1 mL of methanol.

A small fraction of this methanolic solution (50 μL) was used to determine the approximate concentration of the free ligand. The absorbance was taken at 232 nm and an exctinction coefficient of 70000 was used to calculate the concentration.[1] Subsequently a CD solution was prepared (0.3 mM). One equivalent of each $Cu(ClO_4)_2$ and NH_4SCN were added and the CD spectrum recorded.

[1] Canary, J. W.; Allen, C. S.; Castagnetto, J. M.; Wang, Y. *J. Am. Chem. Soc.* **1995**, *117*, 8484.

CD - Data

Amino Acid	CE 1 (mdeg)	Inflection Point	CE 2 (mdeg)	Concentration (mM)
L-Histidine	239 nm (-52)	230 nm	226 nm (+12)	0.35
L-Valine	239 nm (-52)	232 nm	228 nm (+15)	0.31
L-Tryptophan	239 nm (-125)	233 nm	231 nm (+72)	0.34
L-Cysteine	239 nm (-50)	232 nm	228 nm (+28)	0.38
L-Tyrosine	240 nm (-71)	234 nm	231 nm (+12)	0.18
S-Phenylglycine	240 nm (-33)	232 nm	225 nm (+10)	0.30
L-Arginine	239 nm (-28)	233 nm	231 nm (+12)	0.17
L-Leucine	239 nm (-90)	232 nm	230 nm (+24)	0.29
L-Alanine	239 nm (-81)	232 nm	228 nm (+12)	0.15
L-Isoleucine	239 nm (-75)	232 nm	228 nm (+35)	0.35
L-Methionine	239 nm (-33)	232 nm	229 nm (+15)	0.18
L-Serine	240 nm (-25)	233 nm	227 nm (+17)	0.50
L-Aspartic Acid	240 nm (-45)	232 nm	227 nm (+37)	0.90
L-Lysine	239 nm (-30)	232 nm	228 nm (+11)	0.36
L-Phenylalanine	240 nm (-64)	231 nm	229 nm (+23)	0.13
L-Glutamic Acid	240 nm (-27)	232 nm	228 nm (+21)	0.58
L-Asparagine	239 nm (-38)	233 nm	228 nm (+21)	0.40
L-Glutamine	240 nm (-29)	232 nm	226 nm (+12)	0.36
L-Alanine methylester	239 nm (-95)	231 nm	228 nm (+10)	0.18
S-Phenylglycine methylester	240 nm (-35)	232 nm	228 nm (+11)	0.36

Amino Alcohol	CE 1 (mdeg)	Inflection Point	CE 2 (mdeg)	Concentraion (mM)
(S)-Isoleucinol	240 nm (-109)	233 nm	229 nm (+29)	0.29
(S)-(+)-2-Amino-3-cyclohexyl-1-propanol	240 nm (-150)	233 nm	229 nm (+35)	0.27
(S)-Leucinol	240 nm (-98)	233 nm	229 nm (+30)	0.23
(S)-Phenylglycinol	240 nm (-84)	233 nm	229 nm (+30)	0.23
(S)-(+)-2-Amino-1-propanol	239 nm (-130)	232 nm	228 nm (+36)	0.19
(S)-(+)-2-Amino-3-methyl-1butanol	239 nm (-98)	232 nm	229 nm (+27)	0.29
(S)-(-)-2-Amino-3-phenyl-1-propanol	240 nm (-92)	233 nm	229 nm (+24)	0.20
(S)-(+)-2-Amino-1-butanol	239 nm (-90)	233 nm	228 nm (+28)	0.19